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DEVELOPMENT OF DISPERSION STRENGTHENED TANTALUM BASE ALLOY

Fourteenth Quarterly Report

by

R. W. Buckman and R. C. Goodspeed

prepared for
National Aeronautics and Space Administration
Lewis Research Center
Space Power Systems Division
Under Contract (NAS 3-2542)

ASTRONUCLEAR LABORATORY
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FOURTEENTH QUARTERLY PROGRESS REPORT

Covering the Period

February 20, 1967 to May 20, 1967

Prepared for

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ABSTRACT

Development of dispersion strengthened tantalum base alloys for use in advanced space power systems continued as the evaluation of 0.04 inch thick Ta-7W-1Re-1Hf-0.012C-0.012N (ASTAR-811CN; Heat NASV-23) sheet was essentially completed. Ductile-brittle transition temperatures of tungsten inert gas welded ASTAR-811CN were determined as a function of longer post-weld annealing times than previously studied. The results were related to microstructure, phase morphology, and hardness data. Investigation of the effects of grain size on the creep properties of Ta-8W-1Re-1Hf (ASTAR-811; Heat NASV-22) and Ta-8W-1Re-0.7Hf-0.025C (ASTAR-811C; Heat NASV-20) was continued. Initial results show that the creep properties of ASTAR-811C when recrystallized to 0.03mm grain size were independent of the final annealing temperature.

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I. INTRODUCTION

This, the fourteenth and final quarterly progress report on the NASA-sponsored program, "Development of Dispersion Strengthened Tantalum Base Alloys" describes the work accomplished during the period February 20, 1967 to May 20, 1967. The work was performed under Contract NAS 3-2542.

The primary objective of the current phase of this program is the processing and evaluation of 0.04 inch sheet of three compositions which were melted as 60 pound, 4 inch diameter ingots. The compositions were selected for potential sheet and tubing applications on the basis of weldability, creep resistance, and fabricability characteristics.

Prior to this quarterly period, several promising tantalum alloy compositions have been developed which exhibited a good combination of creep resistance, weldability, and fabricability.[1] Three compositions, Ta-8W-1Re-1Hf-0.025C (ASTAR-811C), Ta-8W-1Re-1Hf (ASTAR-811) and Ta-7W-1Re-1Hf-0.012C-0.012N (ASTAR-811CN), were selected for scale up. These compositions were double vacuum, consumable electrode arc melted as 60 pound, 4 inch diameter ingots, which were subsequently processed to 0.04 inch sheet by a combination of forging and rolling. Evaluation of all three compositions has been essentially completed.

During this quarterly period, the evaluation of ASTAR-811 and ASTAR-811CN was completed with the exception of a few remaining creep tests. The effect of post weld annealing for times out to 500 hours at 1800-2600°F on the ductile-brittle transition temperature of ASTAR-811CN was determined. The investigation of the effect of annealing time and annealing temperature on the creep behavior of all three compositions was redirected to study only ASTAR-811C and ASTAR-811.

II. PROGRAM STATUS

A. WELDABILITY

Tungsten inert gas (TIG) welded 0.04 inch thick sheet specimens of ASTAR-811CN were annealed for 16 hours at 1800, 2200, and 2600°F (980, 1200, and 1425°C) and for 500 hours at 2200°F (1200°C). The post weld annealed specimens were tested in bending over a 1t bend radius with the weld bead transverse to the bend axis and the ductile-to-brittle behavior was determined. The data are summarized in Table 1 along with 1 hour post weld annealing data reported previously.[2] The effect of post weld annealing temperature and time on the ductile-brittle behavior of ASTAR-811CN is summarized in the data plotted in Figure 1. As-TIG welded, ASTAR-811CN exhibited a DBTT* of -225°F. Post weld annealing for 1 hour at 1800-3000°F resulted in an increase in the DBTT with the peak increase observed after heating for 1 hour at 2600°F. Increasing the annealing time to 16 hours shifted the aging peak down to 2200°F. This behavior is indicative of an aging-overaging reaction and has been observed previously in tantalum alloys containing a reactive metal and nitrogen addition.[3,4,5] At 2200°F, an aging peak as evidenced by the ductility minima was observed after heating for 16 hours. However after heating at 2200°F for 500 hours, the bend ductility was the same as for as-welded material.

Transverse sections from the post weld annealed specimens were mounted and studied metallographically. A hardness traverse was also made on the metallographically prepared specimens. Again as reported earlier,[2] very little hardness variation was observed over the fusion and heat affected zone and base metal. The hardness as affected by annealing time and temperature is summarized in Table 2. Generally there was a decrease in hardness level of about 15 DPH units for specimens post weld annealed for 1 hour irrespective of the annealing temperature. However, as noted previously there was an increase in the DBTT as the post weld annealing temperature was increased to 2600°F. As has been discussed in the 10th Quarterly Report[3] there is a strengthening reaction associated with the nitride precipita-

*DBTT - Ductile-Brittle Transition Temperature

TABLE 1 - Ductile-Brittle Transition Temperature of Post (TIG) Weld Annealed ASTAR-811CN[a] (Ta-7W-1Re-1Hf-0.012C-0.012N; Heat No. NASV-23)

Post Weld Anneal	Test Temperature °F	Test Temperature °C	No Load Bend Angle (Degrees)	Remarks	DBTT °F	DBTT °C
As-TIG Welded	-250	-157	90	Failure	-225	-143
	-225	-143	91	Bend		
1 Hr. at 980°C (1800°F)	-100	-73	92	Bend	-200	-129
	-200	-129	92	Bend		
	-225	-143	77	Brittle failure in weld		
	-250	-157	44	Brittle failure in weld and HAZ		
	-320	-196	30			
16 Hrs. at 980°C (1800°F)	-100	-73	92	Bend	-200	-129
	-200	-129	92	Bend		
	-250	-157	25	Brittle failure in weld and HAZ		
	-320	-196	12			
1 Hr. at 1200°C (2200°F)	-100	-73	91	Bend	-100	-73
	-125	-87	77	Brittle failure in weld		
	-150	-101	26	Brittle failure in weld and HAZ		
	-200	-129	37			
16 Hrs. at 1200°C (2200°F)	+175	+79	88	Bend	+125	+52
	+125	+52	100	Bend		
	+75	+23	51	Brittle failure in weld and HAZ		
	0	-18	90	Brittle failure in weld		
500 Hrs. at 1200°C (2200°F)	-100	-73	92	Bend	-225	-143
	-200	-129	92	Bend		
	-250	-157	90	Very slight brittle failure in weld		
	-320	-196	90			
1 Hr. at 1425°C (2600°F)	+75	+23	92	Bend	0	-18
	+50	+10	90	Very slight ductile failure in weld		
	+25	-4	90			
	0	-18	90			
	-100	-73	52	Brittle failure in weld and HAZ		
16 Hrs. at 1425°C (2600°F)	+75	+23	92	Bend	-175	-115
	-25	-31	90	Bend		
	-125	-87	91	Bend		
	-225	-143	47	Brittle failure in weld and HAZ		
1 Hr. at 1650°C (3000°F)	-50	-46	103	Bend	-100	-73
	-150	-101	84	Brittle failure in weld		
	-200	-129	9	Brittle failure in weld and HAZ		
	-250	-157	21			

(a) Sheet specimens, 0.04 inch thick, were all annealed for 1 hr. at 1650°C (3000°F) prior to welding.

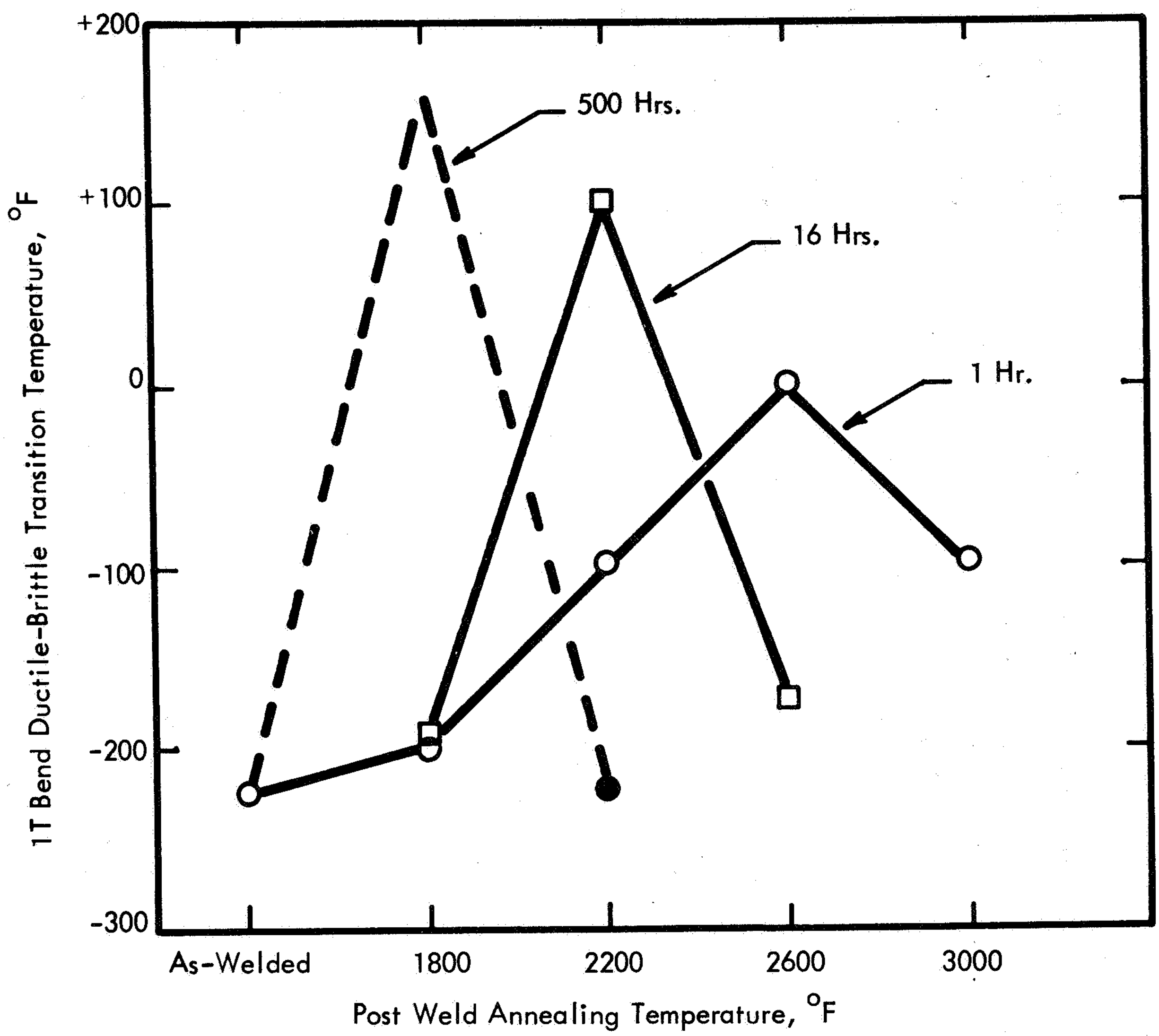


FIGURE 1 - Effect of Post Weld Annealing on the Ductile-Brittle Transition Temperature of TIG Welded ASTAR-811CN

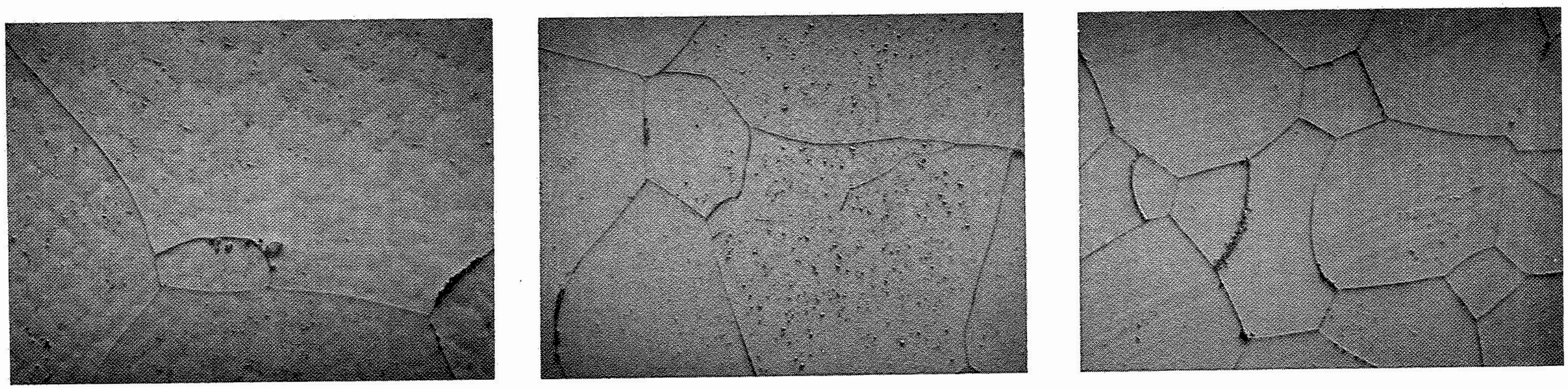
tion and a hardness decrease associated with the carbide precipitation. Both reactions are occurring simultaneously, thus making it difficult to separate the effects of each. Microstructural changes associated with the annealing temperature may explain the ductile-brittle transition behavior,[2] since there does not appear to be a correlation between the room temperature hardness and the ductile-brittle transition temperature.

TABLE 2 - Hardness of Post Weld Annealed ASTAR-811CN TIG Weldments

Annealing Time (hrs.)	DPH After Annealing at Indicated Temperature (°F)				
	As-Welded	1800	2200	2600	3000
1	275	260	260	260	260
16	275	245	245	250	---
500	275	---	220	---	---

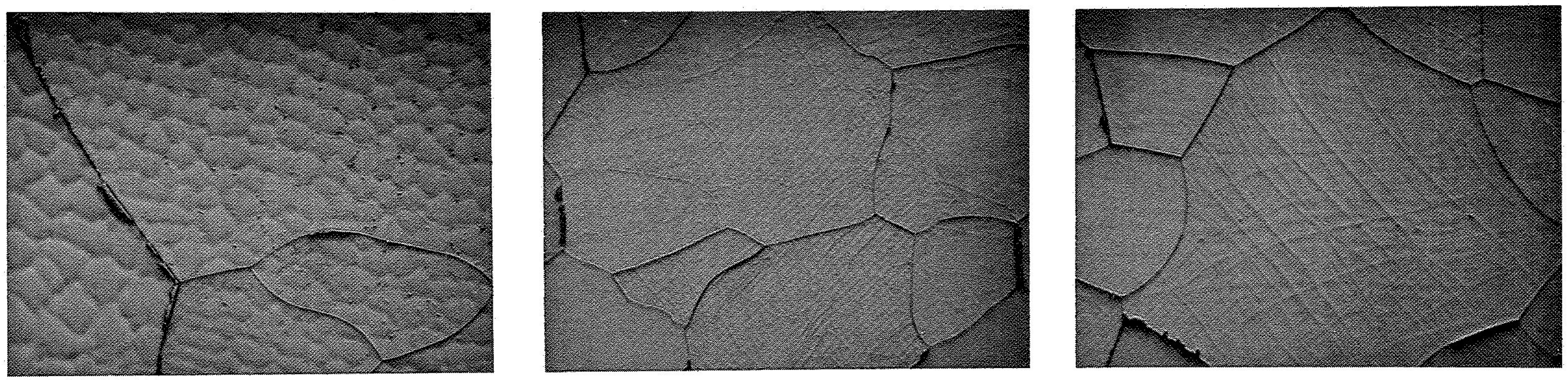
Microstructures of post weld annealed specimens are shown in Figures 2 and 3. The microstructure after post weld annealing for sixteen hours at 1800°F (980°C) (Figure 1) is very similar to that after annealing for one hour at 1800°F. The hardness was also similar and the same ductile-brittle behavior was exhibited after a one and sixteen hour post weld anneal.

The microstructure of specimens annealed for 16 and 500 hours at 2200°F (1200°C) are shown in Figure 3a and 3b respectively. Although there was little observable difference in the microstructure between the specimens annealed 1 hour and 16 hours at 2200°F, there was a significant increase in the ductile-brittle transition temperature, along with a hardness decrease. This behavior for ASTAR-811CN post weld annealed at 2200°F is summarized in Figure 4. Increasing the post weld annealing time to 500 hours resulted in a DBTT of -225°F, (the same as measured for as-welded material)and a resultant room temperature hardness of 220 DPH. As will be discussed under the phase identification section of this report, the precipitate after 500 hours at 2200°F is a Hf(CN) while the Ta_2C (dimetal carbide) is observed after 1 and 16 hours at 2200°F.



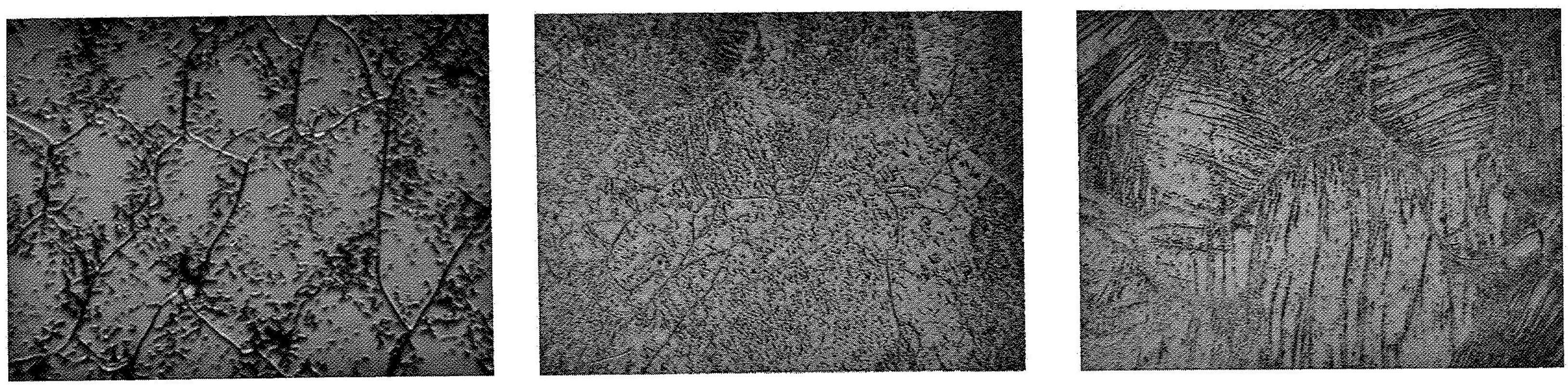
Weld Zone 500X HAZ 500X Base Metal 500X

FIGURE 2 - Microstructure of TIG Welded ASTAR-811CN (Ta-7W-1Re-1Hf-0.012C-0.012N) Specimens After Sixteen Hour Post Weld Anneal at 980°C



Weld Zone 500X HAZ 500X Base Metal 500X

(a) Post Weld Annealed for 16 Hours at 1200°C



Weld Zone 1500X HAZ 500X Base Metal 500X

(b) Post Weld Annealed for 500 Hours at 1200°C

FIGURE 3 - Microstructures of TIG Welded ASTAR-811CN (Ta-7W-1Re-1Hf-0.012C-0.012N) Specimens After 16 and 500 Hour Post Weld Anneals at 1200°C

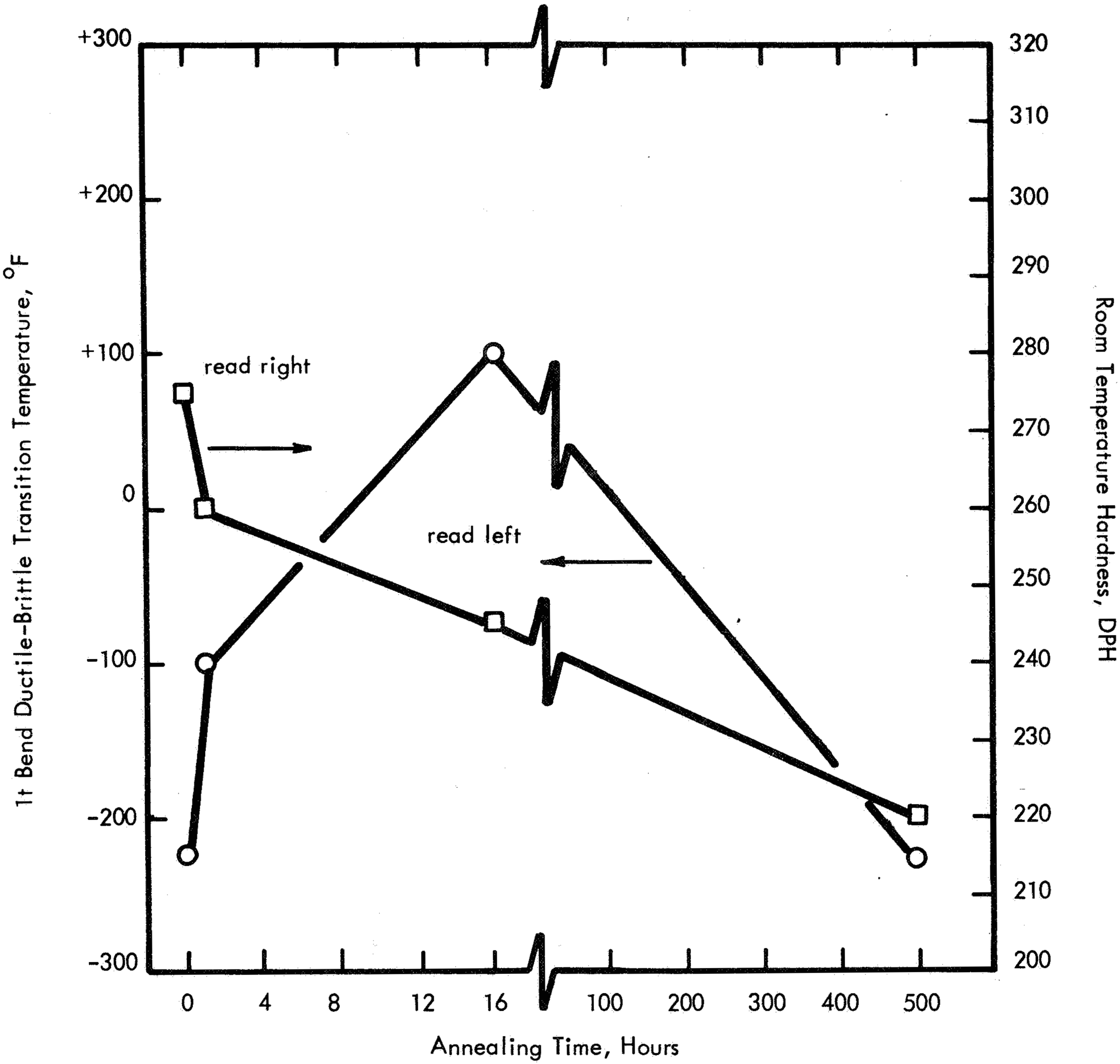


FIGURE 4 - Effect of Annealing Time at 2200°F on the Hardness and Ductile-Brittle Transition Temperature of TIG Welded ASTAR-811CN

The very preliminary results obtained on the influence of post weld annealing time and temperature on the ductile brittle behavior of TIG welded ASTAR-811CN show that the reactions occurring are complex. More detailed study would be required to completely define the specific reactions responsible for the changes in ductility that were observed.

B. EFFECT OF THERMAL TREATMENT ON CREEP BEHAVIOR

Evaluation of the effect of annealing temperature and annealing time on the creep behavior of ASTAR-811C and ASTAR-811 was initiated this period. During the previous quarter, annealing studies had been made on as-rolled 0.04 inch sheet of each composition to determine the grain growth behavior.[2] From these data specimens of ASTAR-811C were annealed for the following times at the indicated temperature to give a recrystallized grain size of 0.03 mm.

Annealing Time (minutes)	Annealing Temperature ($^{\circ}C/^{\circ}F$)
60	1650/3000
10	1800/3270
5	1900/3450
0.5	2000/3630

This is the grain size achieved after heating for 1 hour at 3000°F, and has been the final heat treatment standardized on for creep testing of all the experimental tantalum base alloys. The specimens of ASTAR-811C were then tested in creep at 2400°F and 15,000 psi under ultra high vacuum. The data obtained are listed in Table 3. Also included in Table 3 are the previously reported data obtained on ASTAR-811C which had been annealed for 1 hour at 3000, 3270, and 3630°F (1650, 1800, and 2000°C) prior to testing. These data would tend to indicate that the creep rate is insensitive to the prior annealing temperature if the grain diameter is maintained at a constant size. However, preliminary results indicate that if material is annealed to give a grain size of 0.06 mm the final annealing temperature does appear to be critical.

TABLE 3 - Creep Behavior of ASTAR-811C, Ta-8W-1Re-1Hf-0.025C (Heat NASV-20), Sheet (0.04 Inch Thick) Tested at 2400°F and 15,000 psi

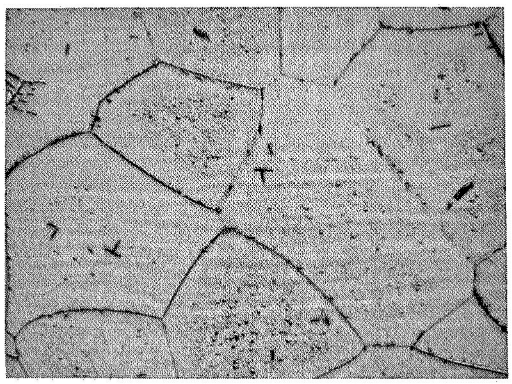
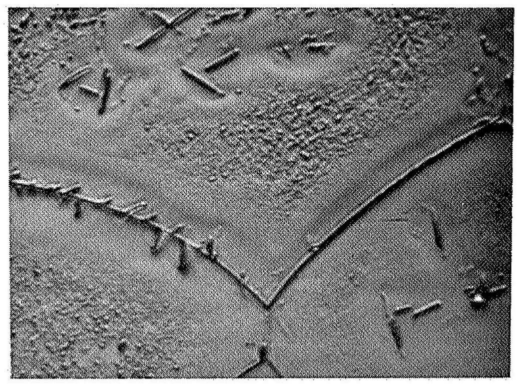
Heat Treatment	Average Grain Diameter[a] (mm)	Test Duration (hrs.)	Total Elongation (%)	Time to 1% Elongation (hrs.)
1 hour at 3000°F/1650°C	0.03	555	2.53	262
10 minutes at 3270°F/1800°C	0.03	472	2.58	241
5 minutes at 3450°F/1900°C	0.03	497	2.12	275
30 seconds at 3630°F/2000°C	0.03	503	2.18	294
1 hour at 3270°F/1800°C	0.06	507	2.00	290
1 hour at 3630°F/2000°C	0.18	1003	2.10	474

(a) Determined by line intercept method.

The behavior exhibited by ASTAR-811C annealed to give a 0.03 mm grain size was unexpected since the as annealed pre-test microstructures were significantly altered by the annealing temperature (see Figure 5). As the annealing temperature was increased from 3000°F to 3630°F, the amount of precipitate visible was significantly decreased indicating that carbon had been taken into solution. The only apparent microstructural difference between material annealed 30 seconds and 60 minutes at 2000°C (3630°F) is the grain size. Otherwise all of the carbon appears to be in solution. However, after testing for 500 hours at 2400°F, the post test microstructures were very similar irrespective of the pretest annealing treatment (see Figure 6). Additional tests are underway on the carbon free analog of ASTAR-811C, ASTAR-811 as well as on specimens which have been annealed to give a recrystallized grain size of approximately 0.06-0.07 mm.

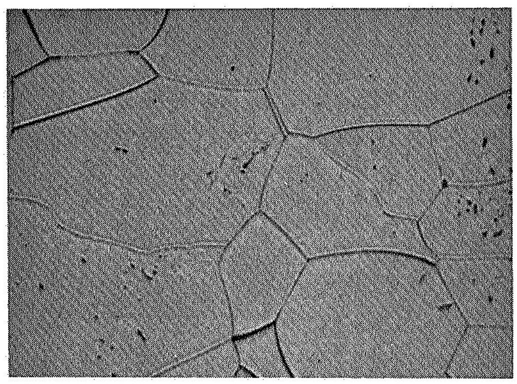
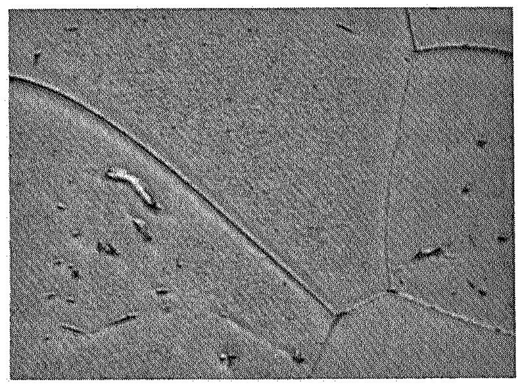
C. PHASE IDENTIFICATION AND MORPHOLOGY

Results reported in the previous quarterly report[2] showed that two precipitation reactions were occurring in ASTAR-811CN. Sheet (0.04 inch thick) which had been given

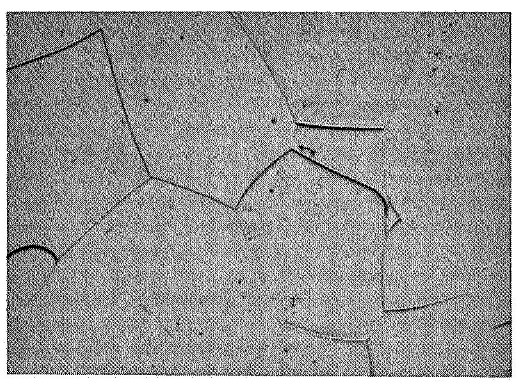
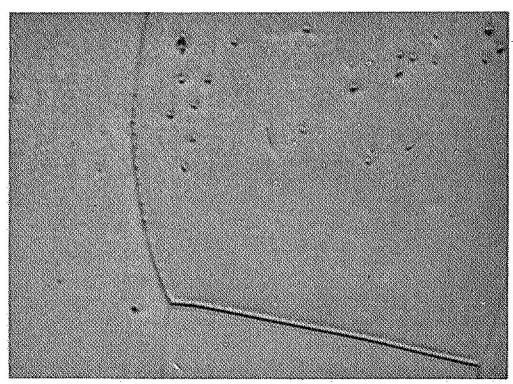
(a) 500X (b) 1500X

1 Hour at 3270°F (1800°C)

(c) 500X (d) 1500X

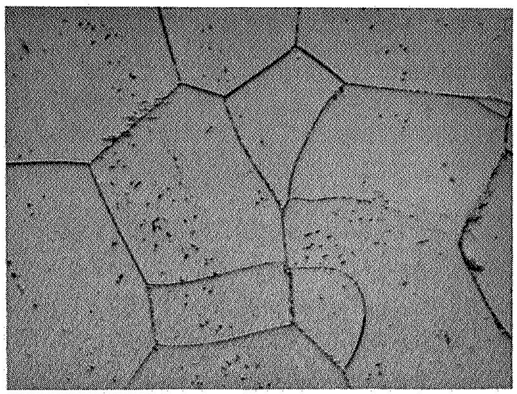
5 Minutes at 3450°F (1900°C)

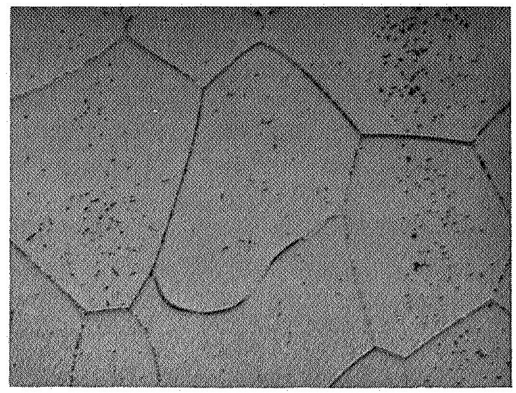
(e) 500X (f) 1500X

30 Seconds at 3630°F (2000°C)

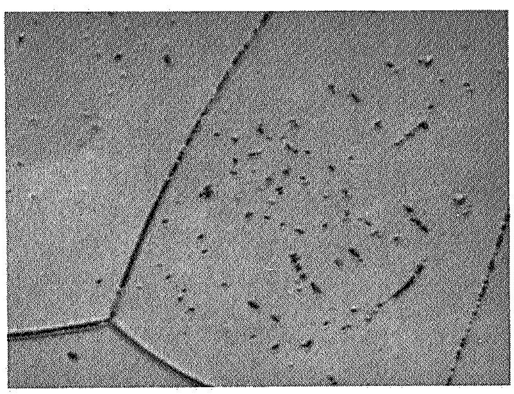
FIGURE 5 - Microstructure of ASTAR-811C (Ta-8W-1Re-0.7Hf-0.025C) as a Function of Pre-Test Annealing Time and Temperature



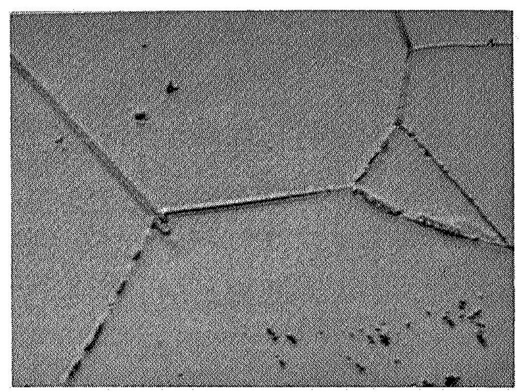
(a) 500X



(b) 500X



(c) 1500X



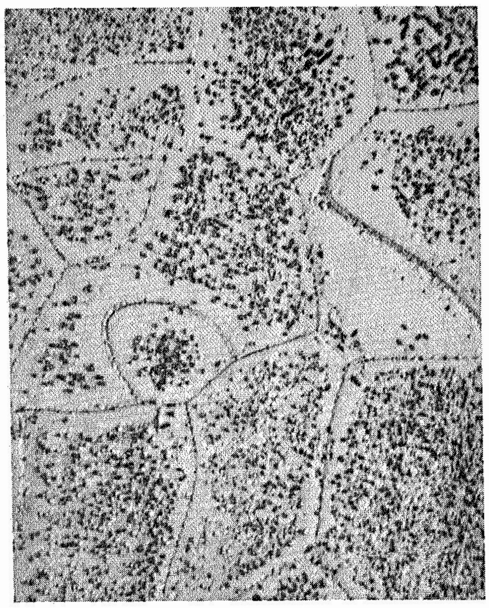
(d) 1500X

FIGURE 6 - Microstructures Representative of Specimens of ASTAR-811C (Ta-8W-1Re-1Hf-0.025C) Annealed 1 Hr./1650°C, 5 Minutes/1900°C, and 30 Seconds/2000°C and Strained 2 to 2-1/2% in Creep Under Stress of 15,000 psi at 1315°C

a 1 hour anneal at 3000°F (1650°C) followed by low temperature annealing treatments at 1800-2600°F for 1 hour resulted in the formation of only a Ta_2C phase. As the annealing time was increased to 16 hours, Ta_2C was still the only phase observed at 1800-2400°F but at 2600°F a Hf(CN) phase was detected.[2] During this period, sheet specimens which had annealed 1 hour at 3000°F (1650°C) were exposed for times up to 907 hours at 2200 and 2400°F. The data obtained are listed in Table 4. Microstructures of the annealed specimens are shown in Figure 7. The microstructure of all four specimens was essentially identical. A fine precipitate existed throughout the matrix and along the grain boundaries. This precipitate was chemically extracted and identified by x-ray diffraction as a FCC phase with a lattice parameter of 4.58 Å. The phase was identified as $Hf_{.75}Ta_{.25}(CN)_{1-x}$. The specimens were also analyzed for carbon, oxygen, and nitrogen and the results are also included in Table 4. There appeared to be a slight decrease in carbon and nitrogen content but more analyses will be required to verify this. The precipitation of the carbonitride phase proceeds in two steps. At 2200 and 2400°F, only the Ta_2C was observed after aging solution annealed specimen for 1 and 16 hours. However, the equilibrium phase apparently is the carbonitride and it will form if the exposure time is sufficiently long. The implications of this precipitation reaction are that the sluggishness of this reaction should improve creep behavior and improved creep resistance has indeed been observed for compositions strengthened with carbonitride phases.[3]

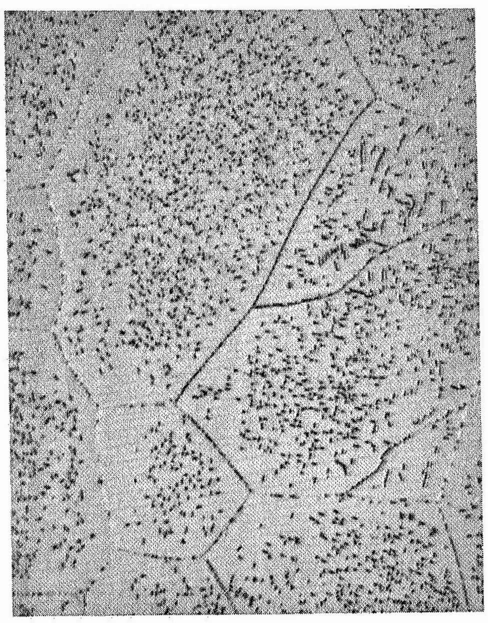
III. FUTURE WORK

During the next period, the remaining experimental work on the effect of annealing time and annealing temperature on creep behavior of ASTAR-811C and ASTAR-811 will be completed. Also a few remaining creep tests will be conducted on ASTAR-811 and ASTAR-811CN. This will complete the experimental work to be done in this investigation. Work will then commence on the writing of a final technical report which is scheduled for publication during the early part of 1968.

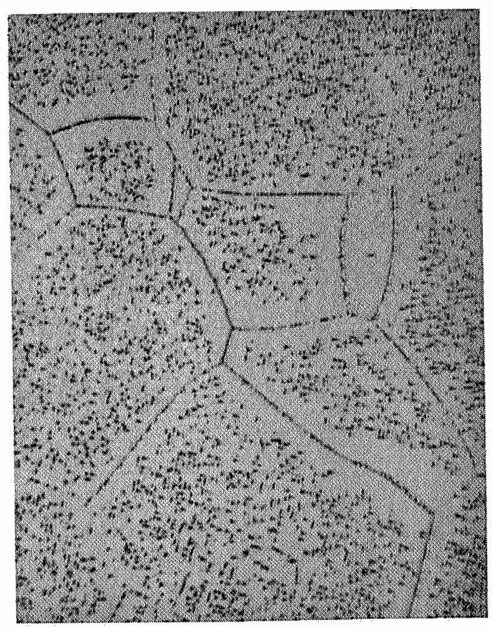


(a) Aged 839 Hrs. at 1200°C

(b) Aged 165 Hrs. at 1315°C



(c) Aged 426 Hrs. at 1315°C



(d) Aged 907 Hrs. at 1315°C

FIGURE 7 - Microstructures of ASTAR-811CN (Ta-7W-1Re-1Hf-0.012C-0.012N) Specimens Annealed for 1 Hour at 1650°C and Aged in Ultra High Vacuum

TABLE 4 - Chemistry and X-ray Diffraction Analyses of Aged ASTAR-811CN (Ta-$7W$-$1Re$-$1Hf$-$0.012C$-$0.012N$) Specimens[a]

Aging Treatment	Chemical Analyses[b] (in ppm)			Bulk Extractions	Diamond Pyramid Hardness[c]
	Carbon	Nitrogen	Oxygen		
839 Hours at 2200°F	110	80	16	FCC; a_o=4.58Å	218
165 Hours at 2400°F	140	100	--	FCC; a_o=4.58Å	229
426 Hours at 2400°F	100	110	--	FCC; a_o=4.58Å	223
907 Hours at 2400°F	94	110	18	FCC; a_o=4.58Å	224

(a) All specimens were annealed for 1 hour at 1650°C (3000°F) prior to aging.

(b) Pre-test analysis C 123 ppm
O 18 ppm
N 125 ppm

(c) Pre-test hardness - 294 DPH

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ERRATA

Please attach the enclosed strip to identify the photomicrographs shown on Page 6 of the Fourteenth Quarterly Report entitled "Development of Dispersion Strengthened Tantalum Base Alloy".

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